WISCONSIN DEPT. OF NATURAL RESOURCES

LABNOTES
Spring 1999

The newsletter of the Wisconsin Laboratory Certification and Registration Program
Program Information: (608) 267-7633 Telefax: (608) 266-5226

Y2K: Will the Millennium Bug Bite You?

Diane Drinkman & Kerilynn Carden, Laboratory Certification Program

As the year 2000 draws near, the laboratory community is faced with many unique challenges to ensure they will not be bitten by the "Millennium Bug." Most laboratories have critical business functions and systems that could experience problems, such as billing and their laboratory information system. As your partner in the laboratory business we urge you to treat the year 2000 issue as a priority among your information technology issues. Laboratories are reminded that Y2K may become a compliance issue, because Chapter NR 149, Wis. Adm. Code, requires that laboratory records, both electronic as well as hard copies, be available for review for three years from the date of analysis.

WHAT SYSTEMS MAY BE AFFECTED IN YOUR LABORATORY?

Laboratory Information Management Systems:

- Log-In Functions
- Worklist Generation
- Hold Time Determination
- Data Entry and Retrieval
- QC Data Collection and Retrieval
- Interfaces with Instrumentation or External Data Systems
- Bar Code Generation and Reading
- Report Generation
- Sample Tracking and Disposal
- Billing

In addition, laboratories must ensure that their LIMS provides correct date sequences and prevents time travel.

General Laboratory Operations: Among those systems that may include data-sensitive microprocessors:

- Climate Control
- Building Security
- Communication
- Data Acquisition modules on autosamplers or coolers

(Please see Y2K on page 2)

In This Issue . . .

LabNotes - Newsletter of the Laboratory Certification Program

LabNotes is published twice annually by the Wisconsin DNR Laboratory Certification and Registration Program. For information about distribution or to make suggestions for future articles, contact the editor.

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(Y2K, continued from page 1)

Instrumentation Concerns: The vast majority of instruments used in laboratories today contain date-sensitive software and/or circuitry that may be affected by Year 2000 problems. Many manufacturers have added Y2K links on their websites to check for compliance.

On-Line Y2K Resources: Many manufacturers of laboratory instrumentation have web sites (on-line searchable databases) that include Y2K compliance status information and assistance. Please consult these sites or contact a member of the Laboratory Certification Program for more information. In addition, the State of Wisconsin has information on-line for municipalities at the Department of Administration homepage (http://y2k.state.wi.us/) and the Federal government's web site (http://www.y2k.gov/) contains a plethora of informaton.

WHAT THE LABORATORY CERTIFICATION PROGRAM IS DOING TO PREPARE

State agencies have been working diligently to minimize the effects of Y2K on day-to-day operations. Within the Department of Natural Resources, software applications are being tested in a "Millennium Room." The Laboratory Certification Tracking System was tested at the end of March and will be fully functional on January 1, 2000

For more information on the Laboratory Certification Program's Y2K efforts, contact Kerilynn Carden at (608) 266-9255 or via email at cardek@dnr.state.wi.us or Diane Drinkman at (608) 264-8950 or via email at drinkd@dnr.state.wi.us.

ADMINISTRATIVE INFORMATION

Changing Laboratory Name or Ownership?

Greg Pils, Laboratory Certification Program

All laboratories about to change their name or ownership are reminded that they must notify the Laboratory Certification Program, in writing, within 10 days of the effective date. If a laboratory maintains 60% of the analytical staff and existing equipment, they must submit a revised application within 30 days of the initial notification. The application, including revised application fee, should note any changes in staffing, methodology, or analytical equipment.

If less than 60% of the analytical staff are retained, the laboratory must apply for certification as if a new laboratory. Name and ownership changes are spelled out in greater detail in s. NR 149.07(7), Wis. Adm. Code. For additional information, contact Greg Pils at (608) 267-9564, or via e-mail at pilsg@dnr.state.wi.us.

Certification and Registration Fees for FY 2000

As anticipated, the Natural Resources Board approved the Laboratory Certification and Registration Program's FY 2000 fees in March. After holding relative value unit (RVU) costs constant from FY 1998 to 1999, the cost per relative value unit will increase from $37.50 to $42.50 for FY 2000. The fees for a typical registered wastewater treatment plant laboratory will increase $70 (base fee + categories 1-4). Commercial laboratories (certified base fee + categories 1-8, 10, 12, & 14-16) will see an increase of $295. Renewal bills will be mailed to laboratories in early May.

ITEM	FY 2000 FEE
Registration, Base	$425.00
Certification, Base	$637.50
Category 1	$42.50
Category 2	$42.50
Category 3	$42.50
Category 4	$42.50
Category 5	$85.00
Category 6	$85.00
Category 7	$170.00
Category 8	$170.00
Category 9	$170.00
Category 10	$170.00
Category 11	$170.00
Category 12	$170.00
Category 13	$170.00
Category 14	$170.00
Category 15	$510.00
Category 16	$170.00
Category 17	$170.00
Category 18	$850.00
Cat. 18 (NO_3 Only)	$85.00
Cat. 18 (NO_3 + F Only)	$170.00
Category 19	$170.00
Category 20	$1105.00
Category 21	$170.00
Initial Application	$255.00
Revised Application	$127.50
Reciprocity	$1275.00

Delinquent Taxes and Dead Beat Dads

When applying for a fishing or hunting license in Wisconsin, the Department of Natural Resources is required to collect the applicant's Social Security number. The following excerpt from the *BEITA Times Newsletter* (March 1999) may help to explain the rationale for collection of this information and how laboratory certification will be affected.

Both the Federal Personal Responsibility and Work Opportunity Act, and the Balanced Budget Act now require states to collect Social Security Numbers (SSN) as a condition of receiving federal funding for child support and family assistance programs. The SSN then becomes the key piece of data in identifying individuals who are delinquent in support payments or who owe back taxes. For that reason these laws are often

referred to as "deadbeat" laws.

Last year, in response to the federal mandate, Wisconsin enacted various State statutes authorizing agencies to collect the SSN from many additional customers including hunters and sportsmen. These new statutes generally specify that this information is to be provided only to the Dept. of Workforce Development.

In the near future, the Laboratory Certification Program will require submission of your federal employee identification number (FEIN), not Social Security Number, to meet this requirement.

Additional information regarding federal and state collection of the SSN appears in Laws Relating to Disclosure of Social Security Numbers, Information Memorandum 98-34, September 8, 1998, from the Wisconsin Legislative Council, at (608) 266-1304. If you have further questions about collection of these identification numbers, please contact your State Senator or Assembly Member

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High Chloride Levels & Nitrate Interference

Greg Pils, Laboratory Certification Program

Laboratories using ion selective electrode (ISE) methods for nitrate testing are cautioned that high concentrations of chloride may impart a positive bias to the analysis, yielding result concentrations that may be as much as double that of the true value.

The use of manufacturer-supplied buffering solutions has proven effective in significantly diminishing the effects of interfering chlorides, if not eliminating them altogether. These buffers are not to be confused with the ion strength adjusters (ISAs) also available, which have proven to be ineffective for removing this particular interference. Labs testing samples for nitrate using an ISE in conjunction with an ISA solution may wish to screen samples for chloride before proceeding with the analysis. For more information, contact Greg Pils at (608) 267-9564 or *pilsg@dnr.state.wi.us*.

Laboratory Certification Program Staffing Changes

Jack Sullivan, Section Chief, Analytical & Statistical Services

In the last few months the Laboratory Certification Program has lost some great staff: Colleen Higgins (West Central Region) Mike Kvitrud (Central Office) and Jeff Ripp (Central Office) have all recently moved on. Colleen, a half-time auditor for the program is returning to school. Jeff, a limited term employee (LTE) for the program, is now working in Washington, DC as a senatorial aide. Mike, also an LTE for the program, has accepted a project position with the DNR Water Supply Program. Colleen, Mike, and Jeff performed their jobs at a very high level and will be missed by all of us. We are moving swiftly to backfill these positions. Your patience in any inconveniences their departure causes is greatly appreciated.

On a more positive note, we have filled two vacancies in the Central Office- the half-time program assistant and LTE chemist. Dan Olson successfully competed for the program assistant position and is already providing a significant contribution to the Program. Dan is usually available from 12:30 – 4:30 so when you call Madison for program information he should be able to assist you. Calls placed at other times will be routed to our phone messaging system and will be handled during Dan's normal working hours. Gül Uludŏgan joined the Central Office staff in late March as an LTE Chemist and will be working with the reference sample program.

1999 Laboratory of the Year Awards Presented

Greg Pils, Laboratory Certification Program

In March, the Natural Resources Board recognized the City of Juneau Wastewater Treatment Plant and Alliant Utilities-Nelson Dewey Station as the 1999 Laboratories of the Year.

Juneau Wastewater Treatment Plant's Ross Canniff accepts the 1999 Small Facility Laboratory of the Year award from DNR Secretary George Meyer.

The **City of Juneau Wastewater Treatment Plant** laboratory was recognized for its efforts as a Small Registered Facility. The laboratory provides analytical support for the city's wastewater treatment plant, analyzing samples for biochemical oxygen demand (BOD) and total suspended solids (TSS).

The laboratory consistently goes above and beyond method requirements to ensure production of high-quality data. For example, *Standard Methods* requires drying TSS samples for at least one hour; the laboratory dries them for two and one-half hours to assure samples are thoroughly dry. Constant weight verifications are performed monthly, rather than the minimum quarterly interval.

In the laboratory's nomination, DNR Audit Chemist John Condron described the laboratory's records as "…highly organized and extensive" and was very impressed that he could not identify one single failure while reviewing the laboratory's last three years' worth of quality control records— a fairly remarkable achievement.

DNR Secretary George Meyer presents the 1999 Registered Laboratory of the Year award to Laurie Vogt (center) and Sharon Klinger-Kingseley (right) of Alliant Utilities-Nelson Dewey Station.

Alliant Utilities-Nelson Dewey Station laboratory, Cassville, was recognized as the Large Registered Facility of the year. The facility provides Hexane Extractable Materials (HEM) and total suspended solids (TSS) analysis for the power plant. DNR Audit Chemist Rick Mealy noted a sense of pride and dedication by the laboratory's staff to produce data of the highest quality.

Laboratory staff encouraged Rick to "…take them through the paces…" so any existing shortcomings could be identified and subsequently corrected. Rick was also impressed with the effort the laboratory put into implementation of the HEM method; one notorious for difficulty in meeting specified performance standards. "Alliant Utilities, " he wrote in his nomination, "can be considered a leader in the successful implementation of HEM testing."

The Laboratory of the Year awards were first presented in 1996, and are intended to recognize those laboratories that have developed exceptional systems for producing high-quality data. Although DNR staff nominated this year's winners, the general public is encouraged to submit laboratories for consideration. To obtain a nomination form, contact Dan Olson by phone at (608) 267-7233, or via e-mail at olsondj@dnr.state.wi.us. Nominations for the 2000 awards are due December 31, 1999.

Municipal Wastewater Laboratory Forum

Rick Mealy, Laboratory Certification Program

QC FAILURES

In many wastewater treatment plant laboratories, quality control failures can be the source of major aggravation. How many times have you read back a set of BOD samples, discovered the GGA was out of control, and you ended up qualifying the whole batch? Perhaps the correlation coefficient of your phosphorus calibration curve is no where near the required 0.995… or the slope of your ammonia probe is consistently less than –54 mV. In all of these cases, you need to take the dreaded "corrective action" to ensure you are generating high-quality data.

CORRECTIVE ACTION

Corrective action is a multi-step process to correct failures in analysis. First, you identify the problem, investigate to determine the cause, make corrections and verify the problem has been corrected. As you work to correct what's wrong, you must document your efforts. For the failures noted above, possible corrective action is indicated below.

If GGA results are consistently above 228.5 mg/L DO you should ask:

- Was the DO meter calibrated properly? If using saturated air for calibration, are you adjusting for barometric pressure?
- Have you changed the seed source? Is the seed correction calculated properly?
- Is there a possibility of nitrification? If your facility has recycle flows, this is a distinct possibility.

After going through these questions, you determine that your barometer has been registering low by checking with the local airport. A Winkler titration confirms this and, five days later, additional GGA standard results average 198 mg/L.

You try and try, but cannot meet the required 0.995 correlation coefficient for phosphorus analysis. Some places to start looking:

- Draw the graph and examine the line. Is there one point that looks way out of line?
- Perhaps the curve drops off at the high end- if so, you may exceed the linear range (about 1 mg/L for most labs).

You quickly determine that the third standard, which is prepared with a serological pipet, looks low. You adjust the concentration so a volumetric pipet can be used, prepare a new curve using six standards and find that R = 0.998.

Finally, you decide to tackle that malfunctioning ammonia probe. You ask:

- Is the membrane intact? Are there bubbles visible?
- Was fresh filling solution used?
- How does the intercept compare to the LOD?

Upon examination of the filling solution, you find that the manufacturer's expiration date was June 1996. A new bottle is opened, the probe refilled and the slope now reads –55 mV.

DOCUMENTATION

Perhaps the single most important part of the corrective action process is documentation. While it is acceptable to document the actions you took to correct your QC failures directly on the benchsheet, laboratories are encouraged to develop a consistent recording format (i.e., a form or logbook) for this information. In the above examples, note that the final step was proof that the initial problem was corrected. Another benefit of good documentation is that there is a "history file" to help analysts with trouble-shooting. With a complete corrective action file you will be able to quickly to explain to your auditor what you did when the GGA results were consistently high, phosphorus calibrations didn't meet correlation criteria and the slope of the ammonia probe didn't meet the requirements of *Standard Methods*.

Total Means Total Recoverable Metals

Tom Mugan, Bureau of Watershed Management

Those of you performing testing for heavy metals in wastewater may have wondered; "What is the difference between 'total copper' and 'total recoverable copper'?" That question has been asked by more than one Department of Natural Resources (DNR) staff person in the years since our toxics control rules took effect in 1989. I can now answer by saying, "There is no difference."

Previous versions of EPA analytical methods distinguished between the two terms in methods for testing heavy metals content in water. The distinction was based on subtle differences in digestion procedures performed on samples prior to the measurement step. However, with the newer methods of measurement currently in use at virtually all laboratories, the various sample preparation (digestion) procedures result in no statistical difference in results.

We have suspected for some time that there remains no basis for distinguishing between total and total recoverable metals. However, until recently, EPA had taken no official position on the issue. An August 1998 memo from William Telliard, Director of Analytical Methods Staff in EPA's Engineering and Analysis Division stated that; "For effluent guidelines, for permitting under NPDES, and for other purposes in EPA's water programs, the terms 'total metal' and 'total recoverable metal' may be used interchangeably to reflect that it is the hard mineral acid digestion procedure that is used."

We have made the decision at DNR to abandon the term 'total metals' and exclusively use the term 'total recoverable metals' (newer EPA methods also use the term total recoverable). Our new permit application only requires testing for total recoverable metals. In the permit documents themselves, we will phase out the term 'total' metals in future reissuances. However, for an interim period, some permits will still contain requirements to test for total metals. If you have a permit written this way, please be aware that we make no distinction between total and total recoverable when these terms pertain to metals (for example, arsenic, cadmium, chromium, copper, lead, nickel, silver, zinc). Future Discharge Monitoring Report (DMR) forms will only have columns for reporting total recoverable metals and instructions will direct permittees to report results as such even if the permit requires testing for total metals.

Conversely, there is a legitimate distinction for 'dissolved metals'. This term should be used when a sample is filtered prior to acid preservation to be analyzed for metals. See the Fall 1998 LabNotes for more information on dissolved-based metals limits and dissolved metals testing. For additional information, contact Tom Mugan at (608) 266-7420 or via e-mail: mugant@dnr.state.wi.us.

QA/QC Training

Rick Mealy, Laboratory Certification Program

During March and April 1999, the Laboratory Certification Program, in partnership with the State Laboratory of Hygiene and Wisconsin Rural Water Association, conducted wastewater laboratory quality assurance seminars in six locations throughout the state.

The need for training was identified through a review of deficiencies commonly cited during on-site evaluations.

Topics discussed included:

- Overview of QA and QC;
- Calibration;
- Detection Limits
- Accuracy (Bias)
- Precision;
- Control Limits;
- Corrective Action; and
- Documentation.

Rick Mealy, Regional Certification Coordinator and George Bowman, Supervisor of Inorganic Chemistry, State Laboratory of Hygiene conducted the seminars.

Attendees have indicated that this was a valuable source of information and have asked the Department to consider offering additional training in the future.

DRINKING WATER INFORMATION

Routine Reporting

Mike Kvitrud, Water Supply Program

The Safe Drinking Water Program recently implemented an automated program to verify laboratory certification status. Failure to report proper information about laboratories performing drinking water analysis can lead to unnecessary problems for your clients. This problem usually occurs when analyses are subcontracted to another laboratory. To prevent these problems:

Use the sampling form provided by WDNR Drinking Water Program. Customized forms are printed for each water system specifying which contaminants are to be monitored for and where samples should be collected. Laboratories should obtain these forms from their clients and use the one for that specific sampling event. If the laboratory does not use the custom forms for reporting, the preprinted information (e.g., Water Supply ID, Entry Point ID, WI Unique Well number, etc.) must be included with the analytical results. Only the analytes specified on that form need to be reported to the WDNR.

Properly identify the laboratory performing the analysis. The laboratory name and identification number should be reported to the WDNR along with the results. If more than one laboratory performs analysis, multiple copies of the form, indicating appropriate laboratory name and identification number should be submitted.

REGULATORY UPDATE

FEDERAL REGISTER

ANALYTICAL METHODS FOR REGULATED DRINKING WATER CONTAMINANTS (DECEMBER 31, 1998) Because EPA received adverse comments to "National Primary Drinking Water Regulations: Analytical Methods for Regulated Drinking Water Contaminants" (September 3, 1998), the direct final rule has been withdrawn. EPA will address comments in a subsequent final action based on the parallel proposal also published on September 3, 1998.

STAGE 1 DISINFECTION BYPRODUCTS RULE: ANALYTICAL METHODS FOR MICROBES, LEAD, AND MAGNESIUM (JANUARY 14, 1999) In this proposal, analytical methods for disinfection byproducts monitoring were identified. For magnesium, the notice proposed six methods: Atomic Absorption (Standard Method 3500-Mg B and ASTM D 511-93 B), Inductively Coupled Plasma (Standard Method 3500-Mg C and EPA Method 200.7) and Complexation Titrimetric Methods (Standard Method 3500-Mg E and ASTM D 511-93 A). The notice also included EPA Method 1001, a Differential Pulse Aniodic Stripping Voltammetry (DPAV) technique for lead. The comment period ended March 1, 1999.

METHOD 1664: HEXANE-EXTRACTABLE MATERIALS (MAY 14, 1999) The long-awaited finalization of EPA's new method for Oil and Grease gravimetric determination was finalized in the May 14, 1999 *Federal Register*.

NATIONAL RECOMMENDED WATER QUALITY CRITERIA (DECEMBER 10, 1998) These standards are used in implementing a number of environmental programs, including setting discharge limits in National Pollutant Discharge Elimination System (NPDES) permits. Water quality criteria are not regulations, and do not impose legally binding requirements on EPA, States, Tribes or the public.

Ignitability

WHICH FLASHPOINT METHOD MAY WE USE?

In the last edition of LabNotes, laboratories were informed of revisions to NR 605, requiring use of method ASTM D-93-96 for flashpoint determinations. Many laboratories have struggled to achieve the ± 1°F tolerance for the reference material, 1,4-dimethylbenzene. Since that time, the Bureau of Waste Management has clarified that equivalent test methods approved by EPA may also be used for flashpoint determinations. This includes ASTM D-93-79 and D-93-80, in addition to D-93-96.

If your laboratory has struggled to meet the ± 1°F tolerance for 1,4-dimethylbenzene, you may use *any* of these methods for ignitability tests. Contact Dave Parsons, WDNR Bureau of Waste Management, at (608) 266-0272 for more information.

WITHDRAWAL OF CYANIDE AND SULFIDE REACTIVITY GUIDANCE

Diane Drinkman, Laboratory Certification Program

The EPA Office of Solid Waste and Emergency Response has identified errors in the development of the guidance and subsequently has withdrawn the methods for Reactive Cyanide and Sulfide as described in Chapter 7 of SW-846. A *Federal Register* notice is forthcoming.

EPA has reiterated that human health and the environment must not be endangered by the evolution of toxic gases when wastes are exposed to specific pH conditions. Generators should utilize knowledge of wastestreams to classify high-concentration sulfide and cyanide-bearing wastes to assign waste codes. For more information regarding withdrawal of cyanide and sulfide reactivity guidance, contact Diane Drinkman at (608) 264-8950 or email at drinkd@dnr.state.wi.us.

PBMS

(POSITIVELY BAFFLING METHOD SURROGATE? OR PANDORA'S BOX MAKES THE SCENE?)

Donalea Dinsmore, Auditor and Quality Assurance Chemist

If you've been ignoring the Performance-Based Measurement Systems (PBMS) initiative until EPA decides how to implement it, your bliss may end soon. The Office of Solid Waste revised SW-846 to explicitly allow method modifications and is working to update approximately 14 regulations to remove the requirements to use of SW-846 methods. The Office of Water is finalizing its Method Flexibility Rule for debut in the Federal Register sometime this summer. The Office of Water modified its original proposal to make EPA approval of new methods optional rather than mandatory, adjusted performance criteria for the inorganic methods, and clarified its requirements for the demonstrations of method equivalency. Rather than defining each effluent or source as a "matrix", the revised proposal uses effluent categories from existing regulations (e.g., metal finisher, pulp and paper, etc.) to define matrix types.

For more information on using PBMS, see Appendix E in Chapter 4 (Quality Systems) of the NELAC requirements which can be downloaded from the EPA web site: www.epa.gov/ttnnela1/.

You may also be interested in ELAB's recommendations for implementing PBMS dated 2/23/99, which is also available from this site. The Office of Water's Guide to Method Flexibility and Approval of EPA Water Methods can be downloaded from: www.epa.gov/OST/Tools/guide/flex.html/

NELAC

NELAC IVi HIGHLIGHTS

Alfredo Sotomayor, Senior Audit Chemist

The fourth NELAC interim meeting was held January 11-14, 1999 in Washington DC. Standing committees typically discuss proposed changes and refine proposals to standards during the interim meeting. The proposals are then presented for ratification at the next annual meeting. But, because standards are fairly complete now, many of the participants focused their attention on the National Program's implementation plans.

TRANSITION COMMITTEE

The Transition Committee gave a progress report, and unveiled tentative plans for recognizing the first accrediting authorities (AA) and for accrediting the first set of environmental laboratories. At the time of the meeting, the National Environmental Laboratory Accreditation Program (NELAP) had reviewed 17 AA applications for completeness and had determined that 13 of them were technically sound. Seven potential AAs had received on-site assessments: California, Colorado, Illinois, Kansas, New Hampshire, New Jersey and New York. Of those, the New York program had been recommended for recognition because the state had addressed satisfactorily all the findings of the on-site assessment. NELAP expected that eight to twelve AA's would be recognized initially. The Transition Committee conducted an informal survey among prospective accrediting authorities and concluded that there would be enough assessors to meet the demand for accrediting interested laboratories.

The timetable presented during NELAC IVi staged the recognition of the first set of AAs by April 1, 1999. These AAs would receive laboratory applications between April and June 1999, and review applications and complete half of the on-site assessments for applicant laboratories between June 1999 and March 2000. By April 2000, AAs would announce the first set of accredited laboratories: some would receive interim accreditation, if they had not been visited on-site, and others would receive full accreditation. The AAs would then complete the rest of the on-site assessments between April 2000 and March 2001.

However, during a February teleconference, the NELAC Board of Directors approved a motion to grant recognition to the first set of AAs at NELAC V in June. The Board thought this would allow these AAs to accredit laboratories according to the 1999 standards. It is unclear how this decision affects the rest of the proposed implementation timetable.

ON-SITE ASSESSMENT COMMITTEE

Analytical method checklists were promised by March, and two generic method checklists are now available in the NELAC Web site (www.epa.gov/ttnnela1/). A version of the basic training course for laboratory assessors is expected to be available by NELAC V.

Other noteworthy items from the interim meeting:

- Establishing a distinction between field measurements and field-testing is under discussion to clarify accreditation requirements for mobile laboratories.
- For multianalyte methods, a list of key analytes to obtain and maintain accreditation may be instituted.
- The effective date of revised standards still needs to be clarified.
- The need and practicality of matrix spikes and matrix spike duplicates continues to be debated.
- The Environmental Laboratory Accreditation Board (ELAB) received and reviewed the final version of the PBMS committee's report. This report is also available in the NELAC Website.

At the local front, we have started discussions with Administration and have a tentative workplan to guide us toward NELAP recognition. For more information on our NELAC-related activities, contact me at (608) 266-9257, or at sotoma@dnr.state.wi.us.

NELAC and Reciprocity in Wisconsin

If Wisconsin is granted recognition by NELAP, any laboratory that we directly accredit will be eligible for accreditation for equivalent tests in any other NELAP-recognized state. At the same time, any laboratory accredited by a NELAP-recognized AA would be eligible for reciprocal accreditation in Wisconsin. We would, like many other states, charge a fee to grant reciprocal accreditation to out-of-state laboratories.

But what would happen to our current reciprocity agreement with various states? Because the agreements we currently have are not based on the NELAC standards, we would not be able to use them for NELAC reciprocity. If we succeed in becoming a NELAP-recognized accrediting authority, we plan to expire all current reciprocity agreements, with prior notice, and to rely on NELAC accreditation to grant reciprocal accreditation. Until such time, reciprocity with the Wisconsin program will remain status quo.

THE AUDITOR'S CORNER

Alfredo Sotomayor, Senior Audit Chemist

How good do you look? It is a truth universally acknowledged that we all make efforts to look our best when under scrutiny. But sometimes good looks can get in the way of seeing the real you, the unadorned, raw you. Mistakes in handwritten documents are often fixed with "white-out", but doing this obliterates an original observation. Data massaging, weighted curves, and that age-defying cream, "Voyage du Temps", sold here as "Time Traveling", all bespeak of a desire to make the rough smooth, the unwieldy pliable, and the messy neat. I call all of them examples of cosmetic chemistry. Lately, you may have heard a lot about peak shaving. This column examines that cosmetic buzz.

THE RAZOR'S EDGE

Peak shaving is the common term for unjustifiably excluding area when integrating a chromatographic peak. This is usually done to comply with quality control acceptance criteria, or to lower an analyte's concentration in a sample. I say "unjustifiably" because chromatography involves judgment from experienced analysts and there are many times when excluding portions of a peak from the analytical signal is the right thing to do. Sometimes, integration software is not sufficiently accurate or discriminating to integrate all peaks defensibly, and analysts must then resort to manual integration.

Almost all of us would agree that cutting a peak in half horizontally, by improper baseline placement, or vertically, by declaring the onset of a peak at its apex is unjustified. But what to do about the in between cases? How can judgment be applied correctly when integrating peaks?

Of course, there are no clear answers to these questions. This is partly because, contrary to expectations, there is no primer on how to integrate signals properly. Protocols that specify the mode of integrating a chromatographic signal, such as our GRO and DRO methods, do so for unique reasons, which are usually not applicable to other tests.

AVOIDING NICKS

The fundamental principle of quantitative integration is that samples should be integrated in the same style chosen for integrating calibration standards. If you really consider this, you will conclude that some integration styles are more prone to error than others. I advise integrating peaks as discrete entities, by connecting the onset of a peak to its end shoulder by a straight line, however inclined the line may be. This used to be called "valley to valley" or "penetrating" baseline by some integrators. I prefer this to the other common integration style of dropping vertical lines that mark the onset and the conclusion of a peak down to a common baseline, because discrete integration excludes baseline rises resulting from sample background, which is not normally found in standards. This gives a "net"

analyte signal in samples, if you will.

If this is to be done electronically, as is now common, peak threshold and area reject parameters must be properly set before a run. But because chromatographic stubble varies within a run, these parameters usually need to be reset as a function of time. Uniformly high thresholds and area rejects miss early eluters, while the converse complicates chromatograms by mistaking noise for signal.

BEFORE AND AFTER SHAVE

All chromatograms must be reviewed by experienced analysts to determine whether peaks have been integrated properly. When there is a need to reintegrate selected peaks manually or to re-establish integration parameters for an entire run, two measures will make your process more defensible: flag any peaks integrated manually or clearly label electronically reprocessed runs; and, retain copies of all runs before they are re-integrated or reprocessed. Unfortunately, this is usually where the paper or electronic trails tend to be deficient. Retaining this information allows others, including auditors, to verify your judgment, and should it become necessary, recompute results using original observations.

COSMETIC SURGERY OR CORRECTIVE ACTION?

When it comes to integration, opt for the latter. When quality control results indicate borderline conditions, take corrective action directed at your analytical system before it deteriorates to the point you feel compelled to perform surgery. No amount of electronic or manual manipulation can substitute for cleanups in dirty extracts, for needed re-tuning, or recalibration. In the data business we must all go beyond what makes us look good into what makes us be right.

IMMUNOASSAY CERTIFICATION

In the administrative code change last year, the Department created a new test category for laboratories performing immunoassay work for a covered program. This category will become effective September 1, 1999. If your laboratory is performing immunoassay testing using SW-846 methods 4010A, 4015, 4020, or 4035 you must apply for certification before the effective date. Please contact Diane Drinkman at (608) 264-8950 or by e-mail at drinkd*@dnr.state.wi.us*.

SPRING MEANS RENEWAL TIME IN WISCONSIN

January	• DNR begins accepting reference sample results.
May	• DNR sends a letter requesting reference sample results. • DNR mails renewal bills to labs.
June	• DNR begins mailing renewal certificates to labs that have paid their renewal fees and submitted acceptable reference sample results.
August	• DNR assesses a late fee to labs which have not paid their renewal bill by August 1.
September	• Renewal period ends September 1. All fees and references samples are due. Labs that have not submitted fees and reference samples will not be renewed after Sept. 1.

RENEWAL REQUIREMENTS- REFERENCE SAMPLES

Many laboratories have inquired about the status of acceptable reference sample providers for the Wisconsin Laboratory Certification Program.

	CATEGORY / TEST	APG	ASI	ERA	NYDOH	SLOH
1	**BOD**	Y	Y	Y	Y	Y
2	**TKN, NH_3 & NO_3**	Y	Y	Y	Y	Y
3	**Total Phosphorus**	Y	Y	Y	Y	Y
4	**Total Suspended Solids**	Y	Y	Y	Y	Y
5	**Total Hardness**	Y	Y	Y	Y	Y
6	**COD, Cl, F, SO_4, Cyanide & Total Phenolics)**	Y	Y	Y	Y	Y
8	**All but Hexalent Chromium**	Y	Y	Y	Y	Y
9	**All**	P	N	P	P	P
10	**VOCs**	Y	Y	Y	Y	Y
11	**Semivolatiles by GC**					
	PAHs	Y	Y	Y	Y	Y
	Phenols	Y	N	Y	Y	Y
	Nitrosamines	Y	N	P	Y	Y
	Haloethers	Y	N	P	Y	Y
	Phthalate Esters	Y	N	P	Y	Y
12	**Semivolatiles by GC/MS**	Y	Y	Y	Y	Y
13	**Extractables by HPLC**					
	PAHs	N	N	Y	N	Y
	Carbamates	N	N	Y	N	Y
	Explosives	P	P	P	P	P
	Diquat & Paraquat	N	N	Y	N	Y
	Substituted Ureas	N	N	Y	N	Y
	Benzidines	Y	N	P	Y	Y
	Other Pesticides	N	N	Y	N	Y
14	**Pesticides**					
	Acid Herbicides	N	Y	Y	Y	Y
	2,4-D and Silvex Only	P	P	P	P	P
	Nitrogen Pesticides	N	N	P	N	Y
	Organophosphorus Pests	N	N	P	N	Y
	Triazine & metabolites	N	N	N	N	Y
15	**GRO, DRO & PVOCs**	N	N	N	N	Y
16	**Organochlorine Compounds**					
	PCBs	N	Y	Y	Y	Y
	Chlorinated Pesticides	Y	Y	Y	Y	Y
18	**Safe Drinking Water—All**	Y	N	Y	N	Y

Y = Reference sample package is available and accepted.
N = Reference sample package either not available or not accepted.
P = Reference sample package may be available and accepted. Check with the DNR before ordering samples.
Safe Drinking Water samples must be graded according to the criteria established in 40CFR 141.23 and 141.24.

SUBSTANCES OF CONCERN AT LOW LEVELS

This list is published as a reminder that laboratories are required to report all data down to their limit of detection (LOD). All results greater than the LOD and less than the limit of quantitation (LOQ) must be reported and appropriately qualified (consult NR 149 for definitions of the LOD and LOQ). Be aware that some programs may require laboratories to report the results for all compounds to be reported down to the LOD, even if they do not appear on this list. Check with your clients to determine what reporting requirements apply. Labs may wish to institute the practice of always reporting all results down to the LOD, thereby avoiding confusion and insuring reporting requirements are always met.

INORGANICS

METALS

Antimony
Beryllium
Cadmium
Chromium (Hexavalent)
Lead
Mercury
Thallium

ORGANICS

ACIDS/PHENOLS

Pentachlorophenol (PCP)

BENZIDINES

Benzidine

HALOETHERS

Bis(chloromethyl)ether

NITROAROMATICS

2,4-Dinitrotoluene
2,6-Dinitrotoluene

ORGANICS, CONTINUED

POLYNUCLEAR AROMATIC HYDROCARBONS (PAH)

Benzo(a)pyrene

PHTHALATES & ADIPATES

Di(2-ethylhexyl)phthalate

NONPURGEABLE CHLORINATED HYDROCARBONS

Hexachlorobenzene

DIOXINS/FURANS

Dioxin (2,3,7,8-TCDD)

PCBS

Polychlorinated biphenyls

CHLORINATED PESTICIDES

DDT and Metabolites
Heptachlor
Heptachlor epoxide
Lindane
Toxaphene

ORGANICS, CONTINUED

CARBAMATE PESTICIDES

Aldicarb

NITROGEN PESTICIDES

Alachlor
Dimethoate
Parathion
Trifluralin

VOLATILES

1,1,2-Trichloroethane
1,1,2,2-Tetrachloroethane
1,3-Dichloropropene (cis/trans)
Bromodichloromethane
Bromoform
Bromomethane
Chloroform
Chloromethane
Dibromochloropropane (DBCP)
Ethylene dibromide (EDB)
Methyl tert-butyl ether (MTBE)
Methylene Chloride
Vinyl Chloride

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